

WHAT IS CLAIMED IS:

1. A method for the purification of the reaction product mixture of at least
5 one alkanolamine and at least one ester and/or fatty natural material, optionally, in the
presence of catalyst, said reaction product mixture containing a significant amount of
residual alkanolamine and/or residual catalyst, which comprises providing said reaction
product mixture, and either simultaneously or sequentially, in any particular order:
 - a) adding at least one non-polar solvent to said reaction product mixture;
 - 10 b) heating said reaction product mixture to a desired extraction temperature;
 - c) providing aqueous salt solution to said reaction product mixture;
 - d) adjusting the pH of the reaction product mixture to below 7.0;
 - e) allowing separation of the reaction product mixture into an organic phase and
an aqueous phase after completion of steps (a)-(d); and,
 - 15 f) removing the aqueous phase to produce a hydroxyalkyl amide composition
with a reduced level of residual alkanolamine and/or residual catalyst.
2. The method of Claim 1 wherein the alkanolamine is of the general formula
RNHR' wherein R is hydroxyalkyl of from 2 to about 10 carbon atoms and R' is
hydrogen, alkyl of from 1 to about 10 carbon atoms or hydroxyalkyl of from 2 to about
20 10 carbon atoms.
3. The method of Claim 2 wherein the alkanolamine is selected from the
group consisting of ethanolamine, propanolamine, isopropanolamine, butanolamine,
isobutanolamine, methylethanolamine, butylethanolamine, diethanolamine,

dipropanolamine, diisopropanolamine, dibutanolamine, diisobutanolamine, and mixtures thereof.

4. The method of Claim 1 wherein the parent acid of the ester contains from about 4 to about 22 carbon atoms and the parent alkanol of the ester contains from 1 to
5 about 10 carbon atoms.

5. The method of Claim 4 wherein the ester is a fatty acid methyl ester or mixture of fatty acid methyl esters.

6. The method of Claim 4 wherein the parent acid of the ester is a fatty acid derived from canola oil, castor oil, cocoa butter, coconut oil, cotton seed oil, olive oil,
10 palm kernel oil, palm kernel (olein), palm kernel (stearine), peanut oil, rapeseed oil, safflower oil, soybean oil, sunflower oil, tall oil, corn oil, butter, lard, tallow, yellow grease, blubber, herring, menhaden, sardine oil, babasso oil and mixtures thereof.

7. The method of Claim 1 wherein the fatty natural material is selected from the group consisting of canola oil, castor oil, cocoa butter, coconut oil, cotton seed oil,
15 olive oil, palm kernel oil, palm kernel (olein), palm kernel (stearine), peanut oil, rapeseed oil, safflower oil, soybean oil, sunflower oil, tall oil, corn oil, butter, lard, tallow, yellow grease, blubber, herring, menhaden, sardine oil, babasso oil and mixtures thereof.

8. The method of Claim 6 wherein the parent acid of the ester is a fatty acid derived from coconut oil and the parent alkanol of the ester is methanol.

20 9. The method of Claim 4 wherein the parent acid of the ester is selected from the group consisting of butyric, caproic, caprylic, capric, decenoic, lauric, cis-9-dodecenoic, myristic, myristoleic, cis-9-tetradecenoic, pentadecanoic, palmitic, palmitoleic, cis-9-hexadecenoic, heptadecanoic, heptadecenoic, steric, oleic, linoleic,

linolenic, ricinoleic, dihydroxystearic, nonadecanoic, arachidic, cis-9, cis-11-eicosenoic, eicosadienoic, eicosatrienoic, arachidonic, eicosapentaenoic, behenic, erucic, docosadienoic, 4,8,12,15,19-docosapentaenoic, docosaheptaenoic, lignoceric, tetracosenoic and mixtures thereof.

- 5 10. The method of Claim 9 wherein the parent alkanol of the ester is methanol.
11. The method of Claim 1 wherein the catalyst is a basic catalyst.
12. The method of Claim 11 wherein the basic catalyst is metal alkoxide and/or metal carbonate catalyst.
- 10 13. The method of Claim 12 wherein the metal alkoxide and/or metal carbonate is selected from the group consisting of sodium methoxide, sodium ethoxide, sodium propoxide, potassium methoxide, potassium ethoxide, potassium propoxide, sodium or potassium butoxide, sodium or potassium pentoxide, sodium or potassium hexanate, sodium carbonate, potassium carbonate, cesium carbonate, calcium carbonate,
- 15 strontium carbonate, barium carbonate and mixtures thereof.
14. The method of Claim 11 wherein the basic catalyst is an organic catalyst selected from the group consisting of tetraalkyl ammonium hydroxide, ethylisopropyl amines, Hunig's Base (diisopropyl ethyl amine), DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), DBN (1,5-diazabicyclo[4.3.0]non-5-ene), guanidine, and pentamethyl guanadine.
- 20 15. The method of Claim 1 wherein the catalyst is present in an amount of from about 1 to about less than 100 wt. percent of catalyst concentrate in solvent.
16. The method of Claim 15 wherein the catalyst is present in an amount of from about 10 to about 50 wt. percent of catalyst concentrate in solvent.

17. The method of Claim 16 wherein the catalyst is present in an amount of from about 20 to about 30 wt. percent of catalyst concentrate in solvent.

18. The method of Claim 13 wherein the metal alkoxide and/or metal carbonate catalyst is present in an amount of from about 0.05 to about 1 wt. percent of the
5 reaction product mixture.

19. The method of Claim 18 wherein the metal alkoxide and/or metal carbonate catalyst is present in an amount of from about 0.25 to about 0.75 wt. percent of the reaction product mixture.

20. The method of Claim 19 wherein the metal alkoxide and/or metal
10 carbonate catalyst is present in an amount of from about 0.40 to about 0.60 wt. percent.

21. The method of Claim 14 wherein the organic catalyst is present in an amount of from about 0.05 to about 1.00 wt. percent of the reaction product mixture.

22. The method of Claim 21 wherein the organic catalyst is present in an amount of from about 0.25 to about 0.75 wt. percent of the reaction product mixture.

15 23. The method of Claim 22 wherein the organic catalyst is present in an amount of from about 0.40 to about 0.60 wt. percent of the reaction product mixture.

24. The method of Claim 1 wherein the non-polar solvent is one or more solvents selected from the group consisting of benzene, toluene, xylenes, aromatic mixed solvents, pentanes, hexanes, heptanes, octanes, nonanes, decanes and petroleum distillate
20 fractions.

25. The method of Claim 24 wherein the aromatic mixed solvents are selected from the group consisting of aromatic 100, aromatic 150, aromatic 150ND, alkyl naphthalenes, cumene, alkyl benzenes.

26. The method of Claim 24 wherein the petroleum distillate fractions are selected from the group consisting of petroleum ether, ligroin, naphthas, gasoline fractions and kerosene and mixtures thereof.

27. The method of Claim 24 wherein the nonpolar solvent is present in an amount of from about 95 to about 5 wt. percent of the reaction product mixture.

28. The method of Claim 1 wherein the extraction temperature is at least about 50°C.

29. The method of Claim 1 wherein the extraction temperature is at least about 60°C.

30. The method of Claim 1 wherein the extraction temperature is at least about 70°C.

31. The method of Claim 1 wherein the salt of the aqueous salt solution is at least one monovalent or divalent salt of mineral acids.

32. The method of Claim 31 wherein the aqueous salt solution is present in an amount of from about 90 to about 0.1 wt. percent of the reaction product mixture.

33. The method of Claim 1 wherein the pH is adjusted to less than about 5.

34. The method of Claim 1 wherein the pH is adjusted to less than about 4.

35. The method of Claim 1 wherein the pH of the reaction product mixture is adjusted to below 7 with at least one acid.

36. The method of Claim 1 wherein the acid used to adjust the pH is at least one acid selected from the group consisting of hydrochloric acid, sulfuric acid, nitric acid, phosphoric acid.

37. The method of Claim 36 wherein the acid is present in an amount sufficient to lower the pH to less than 7.

38. The method of Claim 1 further comprising repeating one or more of the method steps of (a)-(f) at least one time.

5 39. The method of Claim 38 further comprising adding a metal silicate absorbent to the separated organic phase.

40. The method of Claim 39 wherein the metal silicate absorbent is of the general formula $xMO:ySiO_2:zH_2O$ wherein M is any metal of Group IA, IIA or IIIA of The Periodic Table, x and y are in a molar ratio of from about 1:1.5 to about 1:3.6 and z is equal to x.

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41. The method of Claim 40 wherein in the formula of the metal silicate absorbent, x is 1, y is 2.6 and z is 1.

42. The method of Claim 40 wherein the metal in the metal silicate absorbent is magnesium, Ca, Ba, Na, Al

15 43. The method of Claim 41 wherein the metal in the metal silicate absorbent is magnesium, Ca, Ba, Na, Al

44. The method of Claim 40 wherein the metal silicate absorbent is present at a level of from about 20 to about 0 wt. percent of the reaction product mixture.

45. The method of Claim 40 wherein the metal silicate absorbent is present at

20 a level of from about 5 to about 2 wt. percent of the reaction product mixture.

46. The method of Claim 41 wherein the metal silicate absorbent is present at a level of from about 20 to about 0 wt. percent of the reaction product mixture.

47. The method of Claim 41 wherein the metal silicate absorbent is present at a level of from about 5 to about 2 wt. percent of the reaction product mixture.

48. The method of Claim 39 further comprising vacuum stripping residual water from the reaction mixture.

5 49. The method of Claim 46 wherein the vacuum stripping is conducted at from about 50 to about 110°C. and at less than about 100mm Hg until the organic phase is dry.

50. The method of Claim 48 further comprising adding from about .25 to about 2.5 wt. percent of a filter aid.

10 51. The method of Claim 50 wherein the filter aid is selected from the group consisting of Celite[®] 545 (95% SiO₂) diatomaceous earth, diatomite, kieselguhr and soda ashflux calcined.

52. The method of Claim 51 wherein the dried organic phase and filter aid is filtered to obtain a hydroxyalkyl amide composition.

15 53. The method of Claim 1 wherein the hydroxyalkyl amide composition contains any monofunctional or difunctional hydroxyalkyl amide.

54. The method of Claim 53 wherein the hydroxyalkyl amide composition contains from about 10 to about 99 wt. percent of monofunctional or difunctional hydroxyalkyl amide.

20 55. The method of Claim 53 wherein the hydroxyalkyl amide composition contains from about 40 to about 90 wt. percent of monofunctional or difunctional hydroxyalkyl amide.

56. The method of Claim 53 wherein the hydroxyalkyl amide composition contains from about 70 to about 80 wt. percent of monofunctional or difunctional hydroxyalkyl amide.

57. The method of Claim 1 wherein the hydroxyalkyl amide has a level of residual alkanolamine that is not greater than about 0.5 wt. percent.

58. The method of Claim 1 wherein the hydroxyalkyl amide has a level of residual alkanolamine that is not greater than about 0.2 wt. percent.

59. The method of Claim 1 wherein the hydroxyalkyl amide has a level of residual alkanolamine that is not greater than about 0.1 wt. percent.

60. The method of Claim 1 wherein the hydroxyalkyl amide composition that has a level of residual catalyst that is reduced by about at least 50 percent.

61. The method of Claim 1 wherein the hydroxyalkyl amide composition that has a level of residual catalyst that is reduced by about at least 75 wt. percent.

62. The method of Claim 1 wherein the hydroxyalkyl amide composition that has a level of residual catalyst that is reduced by about at least 99 wt. percent.

63. The method of Claim 1 wherein steps (a)-(f) are conducted over a period of from about 30 minutes to about 24 hours.

64. The method of Claim 1 wherein steps (a)-(f) are conducted over a period of from about 4 to about 8 hours.

65. A liquid engine fuel or lubricant composition comprising a major amount of liquid hydrocarbon fuel or lubricant and a minor amount of a hydroxyalkyl amide composition prepared by the method of Claim 1.

66. The liquid engine fuel or lubricant composition of Claim 65 comprising a hydroxyalkyl amide composition containing hydroxyalkyl amide, the hydroxyalkyl amide containing less than 0.5 wt. percent of residual alkanolamine.

67. The liquid engine fuel composition of Claim 65 comprising a
5 hydroxyalkyl amide composition containing hydroxyalkyl amide, the hydroxyalkyl amide containing less than 0.1 wt. percent of residual alkanolamine.